

Oxidation of 9Cr oxide dispersion strengthened steel exposed in supercritical water

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Abstract

The oxidation behavior of a 9 at.% Cr oxide dispersion strengthened (ODS) ferritic/martensitic (F/M) steel exposed to supercritical water at different application temperatures was examined. For comparison, two non-ODS F/M steels HCM12A (~12 at.% Cr) and NF616 (~9 at.% Cr) were also examined. The oxidized samples were characterized using gravimetry, scanning electron microscopy/energy dispersive X-ray spectroscopy, X-ray diffraction, electron back-scatter diffraction and transmission electron microscopy/selected area diffraction. A lower weight gain was consistently observed in 9Cr ODS steel at 500 °C and 600 °C. During exposure, the formation of an internal oxidation layer in the 9Cr ODS steel played a key role in establishing the oxidation behavior. In the 600 °C exposure, grain boundary diffusion of cations may no longer proceed dominantly as it did in the 500 °C exposure. Volume diffusion was likely accelerated and the bulk grains became a more important path for element migration, and the benefit from a small quantity of yttrium (0.28 wt%) in the steel became limited. However, because of the fine grain size of the 9Cr ODS steel, Cr quickly segregated to the ferritic grain boundary region in the internal oxidation layer and especially to the internal oxidation layer/base steel interface, resulting in the formation of Cr-enriched spinel ribbons and a Cr-enriched continuous spinel layer. The microstructure that developed slows down the further diffusion of both cations and anions at 600 °C.

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1. Introduction

Corrosion resistance is one of the key requirements for structural materials to be used in the proposed supercritical water-cooled nuclear reactors [1]. Operating the water coolant in the supercritical state increases the operating temperature and subsequently improves the energy conversion efficiency and fuel usage, and reduces pollutant emissions

[2]. The challenge to using supercritical water is that the oxidation rates are significantly enhanced beyond those seen in temperatures and pressures typically existing in water-cooled reactors [3,4].

As candidate structural material for advanced nuclear energy systems, ferritic/martensitic (F/M) steels with body center cubic structure provide good swelling resistance, low thermal expansion coefficients, and high thermal conductivity. However, their application is limited to temperatures of up to ~600 °C due to the loss of creep strength at higher temperatures. One approach to improve

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creep characteristics at higher temperatures is strengthening by the addition of a fine dispersion of oxide particles in the matrix of the steel. Oxide dispersion strengthened (ODS) steels produced by mechanically alloying with Y_2O_3 dispersion particles have been shown to have higher creep strength compared to traditional F/M steels of similar compositions [5–8]. However, the oxidation behavior of 9Cr ODS steels in high temperature environments, such as supercritical water environment, requires investigation.

The purpose of this study was to investigate the oxidation behavior of a typical 9Cr ODS F/M steel in supercritical water at the different application temperatures being considered. The beneficial effect of yttrium, introduced as a dispersed oxide, on oxidation behavior at different temperatures was examined. For comparison, two advanced non-ODS F/M steels HCM12A (~12 at.% Cr) and NF616 (~9 at.% Cr) were also examined and studied.

2. Experimental details

Bar stock of 9Cr ODS F/M steel (24 mm diameter and 60 mm length) was supplied for this study by the Japan Atomic Energy Agency. The alloy had been annealed at 1050 °C for 60 min, air-cooled, and subsequently tempered at 800 °C for 60 min. The chemical composition of the steel is shown in Table 1. The details of the manufacturing process are described elsewhere [9]. The mean grain size of the tested 9Cr ODS steel was ~500 nm reported in a paper [10]. Two non-ODS F/M steels NF616 and HCM12A were also tested for comparison. Their chemical compositions are also shown in Table 1. The NF616 steel was normalized at 1070 °C for 2 h followed by air-cooling and tempered at 770 °C for 2 h followed by air-cooling. The HCM12A steel was normalized at 1070 °C for 1 h followed by air-cooling and tempered at 770 °C for 7 h followed by air-cooling. The typical mean grain size for both NF616 and HCM12A steels was ~10–20 µm [10].

The as received steels were cut into test samples with dimensions of 31.8 mm × 12.7 mm × 0.5 mm,

polished progressively with finer grit silicon-carbide paper, and then final polished with a 1 µm diamond paste. The corrosion experiments were performed in a natural circulation supercritical water corrosion loop at 360 °C, 500 °C, and 600 °C and 25 MPa with a dissolved oxygen concentration of 20–25 ppb. The detailed construction and system capabilities of this supercritical water loop have been described in a previous paper [11]. The exposure times were 333, 690, 1026 h for the 500 °C test and 333, 667, 1000 h for the 360 °C and 600 °C tests.

After exposure, the extent of oxidation was evaluated by weight change measurement using a Scientech SA-80 Milligram Balance with an accuracy of 0.1 mg. A LEO 1530 field emission scanning electron microscope (FESEM) equipped with energy dispersive spectroscopy (EDS) was used to examine oxide structure in both plan and cross-sectional views as well as to analyze composition across the oxide layer thickness. Electron back-scatter diffraction (EBSD) analysis was performed also in the LEO 1530 FESEM incorporated with TSL orientation imaging microscopy (OIM) system. To obtain geometric exposure of internal oxidation layer, the oxidized sample was mounted on a tripod and carefully polished to remove the base alloy. The observation plane is at a depth of 70–75 µm from the gas/scale interface. Transmission electron microscopy (TEM) cross-section samples of the oxidized regions were prepared by mechanical thinning, followed by argon ion milling to achieve electron transparency. A liquid-nitrogen cooled stage, a low ion-milling current/voltage, and a low sputtering angle were used to keep the sample cool during thinning, to minimize specimen damage. Electron diffraction and diffraction-contrast imaging were conducted in a CM200 TEM operated at 200 kV.

3. Results and discussion

3.1. Oxidation kinetics

Fig. 1 shows the weight gain data of the 9Cr ODS steel after exposure to supercritical water at 500 °C and 600 °C and subcritical water at 360 °C for

Table 1
Chemical compositions of 9Cr ODS steel, NF616 and HCM12A F/M steels (wt%, Bal. Fe)

Alloy	C	Al	Si	P	S	V	Cr	Mn	Ni	Nb	Mo	W	Others
HCM12A	.11	.001	.27	.016	.002	.19	10.83	.64	.39	.054	.3	1.89	N: .063; Cu: 1.02; B: 31 ppm
NF616	.109	.005	.102	.012	.003	.194	8.82	.45	.174	.064	.468	1.87	O: .0042
9Cr ODS	.14	–	.048	<.05	.003	–	8.6	.05	.06	–	–	2	Ti: .21; Y: .28; O: .14

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